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APPLICATIONS OF THERMAL ANALYSIS IN QUALITY CONTROL OF SOLIDIFICATION PROCESSES

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Thermal analysis is widely used to determine solidification characteristics of metals and alloys in various metallurgical processes. Computer-Aided Cooling Curve Analysis (CA–CCA) is the most popular thermal analysis technique because of its ease of use and low cost. This paper discusses the principles of CA–CCA and zero curve calculations. The methods for calculating key solidification characteristics of metals from cooling curves are presented, and their importance in the quality control of manufacturing processes are demonstrated. Examples are presented for cast iron, copper and aluminum alloys.

Keywords: *alloys, aluminum, cooling curves, solidification, thermal analysis*

Introduction

Thermal analysis (TA) is used extensively in the metallurgical, steel and foundry industries, and has a critical role in their daily operations to control the quality of modern materials. Early versions of phase diagrams were generated from data collected by analyzing cooling curves. However, by the 1960's newer experimental methods (diffusion couple experiments, differential thermal analysis and thermodynamic calculations) eliminated the effect of undercooling in many phase diagrams [1]. In the critical Fe–C binary phase diagram, liquidus temperatures were as much as 20°C (36°F) lower by TA than by diffusion couple or Differential Scanning Calorimetry (DSC) [1]. Knowledge of phase diagrams and phase transformations is critical in understanding the structure of metals and alloys, and controlling their solidification and subsequent modification to achieve desired properties and attributes.

Determination of melting points and latent heats are typical applications of DSC, which is more accurate than cooling curve analysis for many materials, including metals and alloys. DSC measures the energy (heat) evolved or absorbed by a sample as it cools, heated, or held at temperature. However, DSC is limited to very small samples, in the milligram range, and by cooling and heating rates. It is expensive, requires technical expertise, and is not suitable for metallurgical or foundry shop floor operations.

The alternative, cooling curve analysis (CCA), is simple, inexpensive, and most importantly suitable for commercial applications. It also provides consistent results. This technique was used for many years in fundamental metallurgical studies and for determining binary phase diagrams. An application of CCA is finding the relationships between cooling curve parameters, melt treatments, alloy composition and properties. Cooling curves can be analyzed by several methods [2–12], a more recent method is Computer-Aided Cooling Curve Analysis (CA–CCA). CA–CCA readily incorporates procedures to evaluate parameters such as total latent heat, fraction solid, etc. for multicomponent alloys from the cooling curve [3, 5, 7, 10, 12]. The determination of 'baseline' or 'zero curve' is critical to cooling curve analysis and is discussed below.

Zero curve

The zero curve is the first derivative of the cooling curve and assumes that no transformation occurs during solidification. It follows the first derivative in single-phase regions, namely above T-liquidus and below T-solidus. Traditional CCA uses a sand cup with a single central thermocouple. The cooling curve can be analyzed by a Newtonian method once the zero curve is generated [6]. Recent investigators incorporated a second off-center thermocouple to give more data on solidification and employed a Fourier method to analyze the data and generate the zero curve [3, 5, 10, 12]. The authors published a detailed description of the two techniques (12). Figure 1 shows the cooling rate, first derivative and zero curves calculated by both Newtonian and Fourier techniques for an A356 Al alloy (Al-7%Si).

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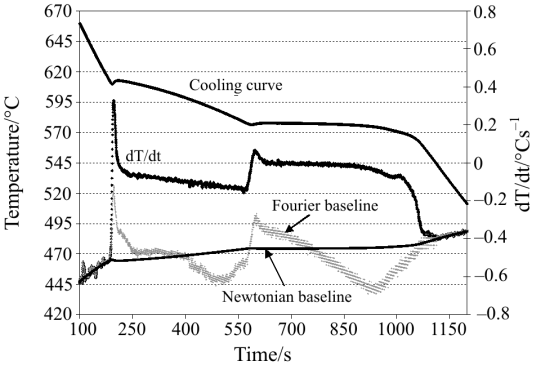

Fig. 1 Cooling curve for A356, first derivative, Fourier and Newtonian zero curves [12]

Fourier and Newtonian zero curves are remarkably different. The predictions by Fourier analysis are expected to be more reliable than the Newtonian method, as it uses the actual temperature field. DSC results can be compared with the calculated latent heats to estimate the relative accuracy of the two techniques.

Latent heat

Latent heat is the energy required to melt or solidify 1 g of a material, expressed in J g^{-1} or kJ kg^{-1}. There is a need for accurate values of latent heats, as published latent heats are only available for pure metals and a few binary alloys [13, 14]. There is no easy way to measure or calculate the latent heats of multi-component alloys that solidify over a wide temperature range. This mushy zone (a region between the liquidus and solidus temperatures) is a changing mixture of liquid and solid phases. Several mechanisms describing the release of latent heat have been used to model casting solidification [15, 16]. These mechanisms use linear and quadratic expressions based on the Lever Rule and Scheil Equation. Most models assume the latent heat released is proportional to the increase in the solid fraction.

The total latent heat, L, can be calculated from cooling curves using the first derivative and Newtonian zero curves (Z_N) as:

$$L = C_P \int_{t_s}^{t_e} [(\frac{dT}{dt})_{cc} - Z_N]dt \qquad (1)$$

where times t_s and t_e are the start and end of solidification, C_P and dT/dt are the specific heat and first derivative of the recorded cooling curve, respectively [12]. The latent heat of solidification of the test sample is simply C_P *x* (area between derived cooling curve and zero curve). Latent heat can be calculated provided the specific heat (C_P) of the test sample material is known. The thermal mass of the crucible should be as small as possible for an accurate estimation of the latent heat. The cooling process should truly reflect the behavior of the solidifying test sample, not the crucible-sample thermal system. The thermal mass of many crucibles are less than 1% of the sample mass.

Similarly, the latent heat can be calculated from the Fourier zero curve (Z_F) as:

$$L = \int_{t_s}^{t_e} \left(\frac{\partial Q}{\partial t}\right)(t)dt \text{ where}$$

$$\frac{\partial Q}{\partial t} = C_V \left(\frac{\partial Q}{\partial t} - Z_F\right) \text{ and} \qquad (2)$$

$$C_V(t) = C_{Vl}[1 - f_S(t)] + C_{VS} f_S(t)$$

where C_V is the volumetric specific heat of the system at any time, C_{Vl} and C_{VS} are the volumetric specific heats of liquid and solid, respectively [12].

Figure 1 shows that the latent heat of an alloy evaluated by the Newtonian procedure (area between the cooling and zero curves) is too dependent upon the fitting method, and therefore, is not a reliable estimate of latent heat. Table 1 shows the total latent heats calculated for an A356 alloy (Al-7%Si-0.33%Mg) by Fourier and Newtonian methods in Fig. 1. The latent heat measured by DSC is included for comparison. Table 1 indicates that the Fourier analysis prediction of the latent heat is more accurate, and therefore, more reliable than the Newtonian method.

Table 1 Latent heat of solidification calculated by Newtonian and Fourier methods using a steel cup, and measured by the DSC method

Alloy	Latent heat/Jg^{-1}		
	Newtonian	Fourier	DSC
A356 (Al-7 %Si)	403	435	432

Solid fraction

A knowledge of fraction of solid at different stages during solidification is critical to the success of some casting processes, e.g. semi-solid casting. The solid fraction at various stages of solidification can be calculated from the cumulative area between the first derivative and zero curves as a fraction of the area between these curves (Fig. 1). The Newtonian method can be used to calculate the solid fraction without the need for the thermo-physical properties such as specific heat. Figure 2 compares the solidified volume fractions predicted by Newtonian and Fourier methods, and shows there are small differences in calculated amounts of fraction solid, especially between the liquidus and Al-Si eutectic temperatures. These differences become less significant as the melt cools

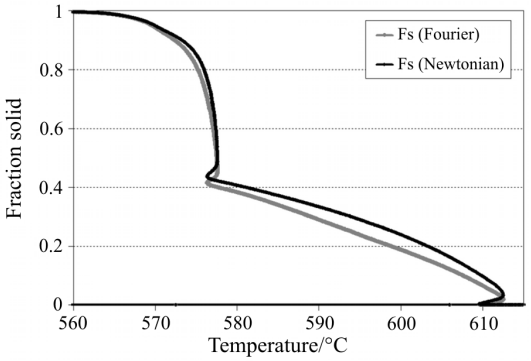

Fig. 2 Comparisons of solidified volume fractions calculated by Newtonian and Fourier methods for the Al–7%Si alloy in Fig. 1 at a cooling rate of 0.55 °C s^{-1} [12]

towards the solidus temperature. Too few cooling curves have been processed and the small differences did not differentiate which method was more accurate for Al–Si alloys. Chen *et al.* found the solid fractions by the two methods were significantly different for eutectic cast iron [6].

Dendrite coherency point (DCP)

The dendrite coherency point (DCP) refers to the state of cast alloy at which a coherent dendritic network is established. Knowledge of DCP is essential in semi-solid casting of metals and eliminating hot tearing. Mechanical, i.e., rheological, techniques and TA are the two main approaches to detect DCP. Bäckerud *et al.* [7] used the two-thermocouple technique to estimate the DCP at the point where the temperature difference recorded between the wall and the center is smallest (Fig. 3).

The DCP is an important characteristic of as-cast alloys because it marks the transition from mass to interdendritic feeding during solidification [7, 17]. Casting defects, such as macrosegregation, shrinkage porosity and hot tearing develop below the DCP [18].

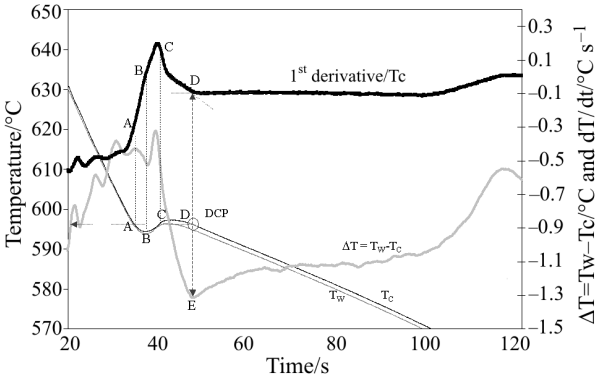

Fig. 3 Cooling, first derivative and ΔT curves for the 319-aluminum alloy (Al-7.4%Si-3.4%Cu-0.35%Fe-0.32%Mg). ΔT is the temperature difference between the wall (T_w) and the center T_C

Therefore, a thorough understanding of solidification behavior at the DCP and the factors that influence it, are crucial for developing new commercial alloys. Although the DCP is a physical phenomenon, its direct detection is virtually impossible. Therefore, indirect methods such as CA–CCA are used to determine the DCP.

Solidification characteristics

Cooling curves are also used to determine critical solidification characteristics of alloys. Some important parameters are liquidus, solidus and formation temperatures of various phases during solidification. These temperatures are readily obtained from the first derivative of cooling curve. Furnace and pouring temperatures are usually set ~100°C degrees above the liquidus temperature. The solidus must be known for castings to be safely removed from the mould. Some castings are heat treated for superior performance. Shorter heat treatment cycles and reduced costs are possible, if castings can be moved to a solutionizing furnace at a temperature just below the solidus.

Most iron foundries adopted TA 30 years ago to estimate the composition of molten irons, as alternative technologies were too expensive, complicated, and took too long. The temperature and height of peaks in the first derivative curve can be used to estimate chemical composition. The first derivative curve is also used to determine the formation temperature and volume fraction of phases during solidification. This is essential as some phases significantly affect a casting's mechanical properties. For example, Fe-intermetallics in as-cast Al–Si–Mg alloys form an α-iron ($Al_{15}(Fe,Mn)_3Si_2$) compound with a 'Chinese script' morphology or thin platelets of β-Al_5FeSi intermetallic. The platelets are more detrimental to mechanical properties. TA elucidates how casting parameters, such as cooling rate, alloying elements and melt treatment, affect the formation of iron intermetallics.

Knowledge of temperature and cooling rate (in the liquid, mushy zone, and solid states) at different locations of the castings is crucial to producing sound engineering castings. The higher cooling rates can cause finer microstructures and better mechanical properties. Cooling curves at different locations in the castings are also needed to check the accuracy of casting simulation software for mould filling and solidification. Undercooling (ABC in Fig. 3), extent of the eutectic plateau, and shape of the cooling curve are used for quality control in the metallurgical industry.

Examples

Ferrous and non-ferrous foundries use different definitions of quality in as-cast products. Aluminum

foundries define quality in terms of chemical analysis, efficient melt treatment, i.e., gas levels, modification and/or grain refinement, percentage of each phase, grain size, etc. Cast iron foundries define soundness as appropriate pouring temperature, chemical analysis, effective inoculation, reduced tendency for chill or shrinkage, and appropriate nodularization in the case of ductile iron.

Traditional practice held that chemistry of the molten metal was sufficient to meet the metallurgical requirements of the cast products. Although chemistry influences metallurgical parameters, as in the case of segregation, chemistry alone is not sufficient and other tools besides spectrometers are required for a melt to meet the specifications. The four cases below illustrate some of the many uses of TA in metallurgical applications.

Aluminum alloys

TA is used extensively in the aluminum casting industry as a quality control tool to estimate the melt chemistry (silicon, iron and magnesium) and to control grain refinement and strontium modification in Al–Si alloys. Strontium is added to change the shape of silicon from needles/flakes to fibrous to improve mechanical properties. Optimum strontium levels are 100–200 ppm to balance benefits and a deterioration of the properties from the formation of secondary phases and increased porosity. The authors have shown that Sr not only reduces the eutectic temperature but also extends the length of the eutectic plateau and changes the shape of the cooling curve (Fig. 4) [19, 20]. As these two parameters are readily obtained from the cooling curve, TA can estimate the Sr level in the melt without the need for timely and costly analytical techniques, such as spectrometric or wet analyses.

Grain size is another important characteristic of an Al casting. Finer grains in Al alloys not only improve properties but also prevent cracking during casting. In hypoeutectic Al–Si alloys, the most important group of aluminum foundry alloys, the addition of Ti, B or Ti–B master alloys reduces grain size, but the concentration of grain refiners should be controlled. Too much or too little can adversely affect the properties, and they are expensive. In hypereutectic Al–Si alloys, the effect of inoculation depends on the size and number of the primary silicon particles [21]. Phosphorus also refines the silicon particles and redistributes them. Figure 5 shows TA can estimate the level of inoculant [Ti- or P-based] in the melt by the under-cooling and slope of the eutectic plateau [20]. Figure 5 also shows increasing the TiB levels shifts the cooling curve upwards and reduces the undercooling [22].

Cast irons

Thermal analysis is used in iron foundries as a quality control tool to evaluate carbon, silicon, and carbon

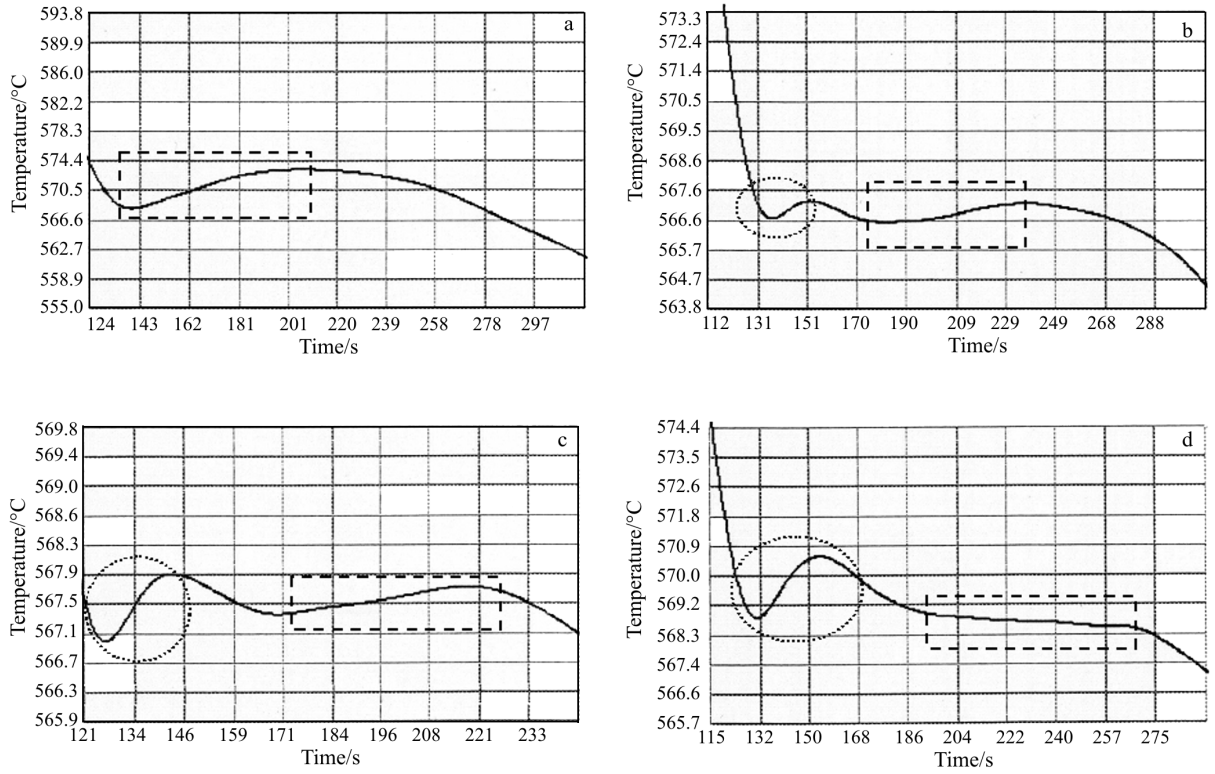

Fig. 4 Cooling curves for a – 0.03%, b – 0.05%, c – 0.075% and d – 0.1%Sr additions to Al–12–13%Si [20]

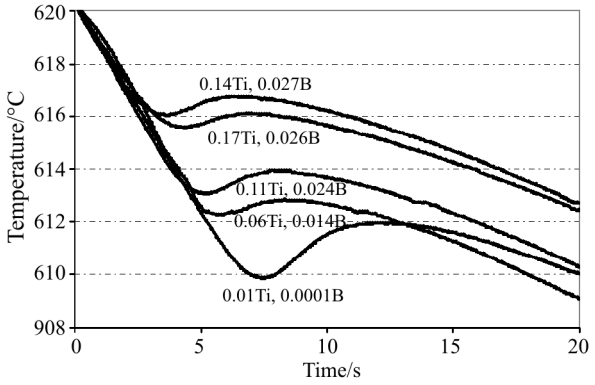

Fig. 5 Effect of Ti and B content (in mass %) on the cooling curve for A356 alloy (Al-7%Si) [22]

equivalent on the melt deck, to forecast the final structure including morphology of the graphite and iron matrix, and to predict mechanical properties as well as properties such as machinability.

Thermal analysis has been used on the foundry floor to analyze liquid irons since BCIRA used TA to measure Carbon Equivalent (CE) in 1961 [23,24]. Subsequent BCIRA research led to a reliable calculation of the carbon content of irons, which helped Leeds & Northrup introduce the first BCIRA carbon calculator (Fig. 6). These were replaced by microprocessors to analyze many types of iron, and as chemistry is displayed directly, they are used on the melt deck to make adjustments. These TA units may be the only quality test used in smaller iron foundries.

Fig. 6 Two different carbon equivalent calculators

Carbon and silicon content can be estimated from cooling curves when iron is solidified in a tellurium coated sand cup (the tellurium causes the iron to solidify as white iron rather than gray iron). Silicon is a major alloying constituent in cast irons, and is incorporated into the austenitic carbon equivalent, raises the graphite eutectic solidification temperature, and lowers the carbide eutectic range. Equation (3) relates the austenite liquidus temperature (T_{AL}) of the ductile base iron to C and Si, while the carbide eutectic temperature (CET) in Eq. (4) is used to calculate the %Si.

$$T_{AL} = 0.556\ (2962–212.3\ (\%C+0.25\%Si))\ \text{in°C} \quad (3)$$

$$\text{CET} = 2085.4–22.7\%Si \quad (4)$$

Once the eutectic composition is determined, the silicon and carbon compositions can be obtained from the expressions:

$$\%Si = (2085.4–CET)/22.7 \text{ and}$$

$$\%C = (2962–1.8T_{AL}–53.07\%Si)/212.3 \quad (5)$$

Although actual equations vary for the range of carbon, silicon and iron type, thermal analysis produces accurate estimations of the %C, %Si and CE of the iron [1, 23–26].

Cooling curves also respond to the carbon morphology, and can predict the cast structure of the molten iron while still in the furnace. Figure 7 shows the cooling curves of the important cast irons: gray, ductile and vermicular. These cooling curves show that irons with similar compositions of the major constituents (C and Si) and appropriate levels of minor constituents will solidify as different structures with different cooling characteristics and different critical points. Most gray irons are hypo-eutectic and start to solidify by the nucleation of primary austenite dendrites, but their shape and size influence the type of graphite formed. Studies showed the shape of the cooling curve reflects the solidification process of the molten iron in the sam-

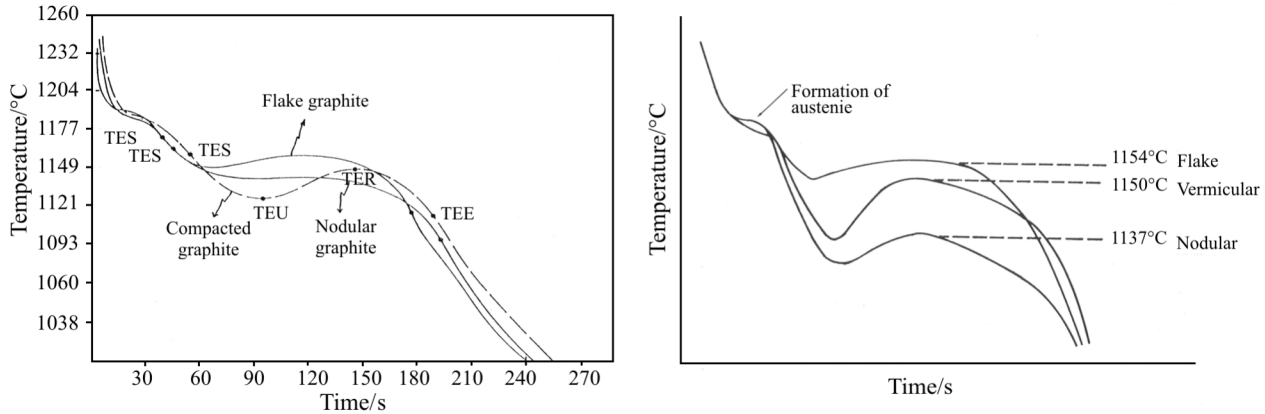

Fig. 7 Schematic cooling curves for gray, ductile, and vermicular graphite irons [6, 29]

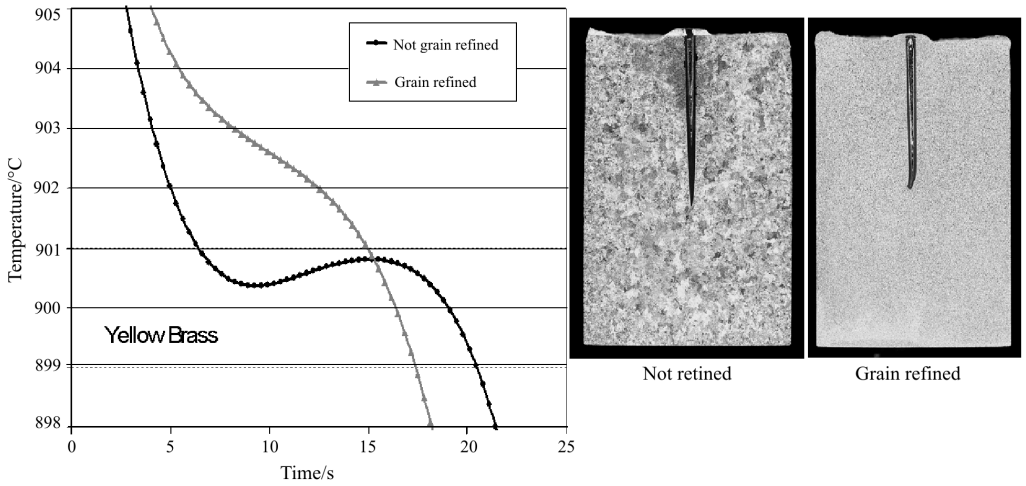

Fig. 8 Effect of grain refiner on cooling curve and on casting structure [36]

ple cup, and developed relationships to predict the as-cast graphite morphology and their amounts (impacts mechanical properties) from critical points on the cooling curves [6, 25, 26]. Stefanescu *et al.* were able to demonstrate the effect of magnesium treatment and post-inoculation (both necessary to produce ductile iron) on the maximum temperature of eutectic arrest (TER) and the eutectic undercooling temperature (TEU) [6]. While later studies tended to differ on their significance, it was not until 1990, that Bradley *et al.* found that size, geometry, and insulation in different commercial cups affected the results (particularly the derivatives) [27]. These cups plug into a stand and are made of sand with an embedded thermocouple. Zhu and Smith found TA was more sensitive to which nodulariser was used to produce ductile iron and their interactions with sulfur [28].

The inter-relationship between the properties of cast irons (strength, structure, fluidity, hardness, chill) and carbon equivalent or chemical composition was recognized many years ago. These relationships took the form of nomographs or equations, which were later related to casting characteristics such as cross-section [30–32]. It was not until Glover *et al.* realized that TA offered expressions for carbon equivalent, composition, and microstructure that predictive relationships using TA were explored using data collected at two foundries [33]. Glover concluded that data loggers of that era lacked the sensitivity to make statistically significant predictions. Furnace atmosphere is another factor that was not considered in their study. Strong found that the constant in Eq. (1) increases by 4% (2% down and 2% up) and the slope by 10% as the atmosphere goes from reducing to oxidizing [34]. Although recent studies have had some success in predicting hardness for different section sizes for specific types of iron, the complex relationships include chemistry (at least 7 ingredients), cooling rate, and cooling curve characteristics [35]. Other more complex analyses have tried to predict other properties but their application was limited to the original conditions.

Permanent mold casting of copper alloys

TA can be used to predict whether liquid metal in the furnace will produce grain-refined castings in the plumbing alloys: leaded yellow brass C85800 and its lead-free counterpart EnvroBrass III C89550 [36]. Grain refinement in these alloys improves casting characteristics, reduces hot tearing, improves mold filling and pressure tightness of plumbing components. These characteristics reduce the number of defective castings and their associated costs. Grain refiners, such as boron, reduce grain size by several orders of magnitude. Adequate levels of grain refiners manifest their presence by the suppression of under-cooling. Figure 8 shows that grain refinement eliminates undercooling. TA quickly indicates the level of grain refinement even as their effects fade from oxidative losses, or by dilution from addition of fresh ingot or returns to the furnace.

Primary aluminum smelting

TRIMET ALUMINUM AG smelter is using TA to control material and thermal balances and stabilize electrochemical cell operations of Hall-Héroult cells [37]. Alumina and cyrolite are fed onto the crust over the two liquids (salt and metal). Bath composition determines the liquidus temperature, and superheat controls the thickness of the crust and dissolution of alumina. The freezing and melting of the crust is affected by energy input and material balance. The new control procedure reduces the liquidus and bath temperatures, as well as superheat, without de-stabilising the pot. Improved operating, energy and cost efficiencies

result from reduced cell voltages and increased materials throughput.

Liquidus temperature and superheat were traditionally estimated from bath temperatures, and XRD analyses of bath constituents. Delays in analyses, sampling errors (structural changes to the frozen sample as well as incomplete analyses), mismatch of temperature and bath sample locations, and finally calculating superheat from theoretical liquidus equations, led to delays and errors controlling the pots. Superheats calculated from theoretical liquidus equations were sometimes negative. The new technique uses TA to determine the bath and liquidus temperatures directly from a small, cooling sample of the cryolite bath [37, 38]. A disposable sensor to measure bath temperature and cathode voltage drop can also monitor sludge formation. The information is used to adjust voltage and cryolite addition and maintain bath temperature, superheat and cell voltage within predefined control limits.

Conclusions

Computer-aided cooling curve analysis is an inexpensive, simple and rapid procedure that finds many applications in the metallurgical and foundry industries. The solidification characteristics of metals obtained from cooling curves can be used to predict cast structures, extent of modification and grain refinement, but is limited to specific situations in making predictions of cast properties. CA–CCA can be used to calculate latent heat, solid fraction and dendrite coherency point from the cooling curves of multi-component alloys. A critical issue is the determination of the zero curve. The Fourier method is more complicated, but more accurate than the Newtonian method.

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